

(*R,S*)-1,2-Bis(benzylsulfinyl)ethane**Jian-Rong Li, Ruo-Hua Zhang
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Key indicators

Single-crystal X-ray study

 $T = 293\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$ R factor = 0.040 wR factor = 0.130

Data-to-parameter ratio = 14.6

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The molecular structure of the title compound, $\text{C}_{16}\text{H}_{18}\text{O}_2\text{S}_2$, adopts the *R,S* form, and has a center of symmetry at the midpoint of the central C—C bond. All the C and S atoms between two phenyl rings are coplanar; this plane is perpendicular to the planes of the phenyl rings. The two S=O groups lie on opposite sides of that plane and their pseudo-torsion angle ($\text{S}=\text{O}\cdots\text{S}=\text{O}$) is 180° , as required by symmetry.

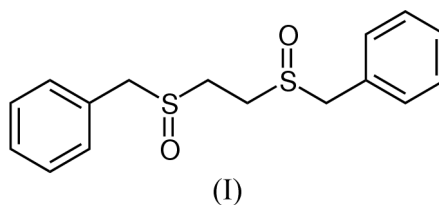
Received 22 April 2002

Accepted 12 July 2002

Online 25 July 2002

Comment

Disulfoxides are known to act as multifunctional ligands, which coordinate to metal ions *via* either their O or S atoms, according to electronic and steric factors. The inherent chiral properties of the S atom produce the diastereomeric *meso* and *rac* forms. Compounds of the type $RS(\text{O})(\text{CH}_2)_n\text{S}(\text{O})R$ exist as diastereoisomeric *rac* and *meso* forms which have different melting points. X-ray crystal structure analyses of the disulfoxides ($R = \text{methyl}, n\text{-propyl}$ and phenyl) proved that the higher melting isomer is the *meso* form and the lower is the *rac* form (Svinning *et al.*, 1976; Shao *et al.*, 1989; Cattalini *et al.*, 1979). As part of our systematic investigation of the coordination chemistry of disulfoxides, we recently synthesized a *meso* form of 1,2-(benzylsulfinyl)ethane, (I). Here, we report the synthesis and crystal structure of (I).



The structure of (I) (Fig. 1) has the *R,S* configuration. The molecule has a center of symmetry at the midpoint of the central C—C bond. A similar situation was found in (*R,S*)-1,2-bis(methylsulfinyl)ethane (Svinning *et al.*, 1976) and (*R,S*)-1,2-bis(phenylsulfinyl)ethane (Cattalini *et al.*, 1979). All the C and S atoms between the two phenyl rings are coplanar; this plane is perpendicular to the planes of the phenyl rings, which are parallel to each other. The two S=O groups lie on opposite sides of that plane and their pseudo-torsion angle ($\text{S}=\text{O}\cdots\text{S}=\text{O}$) is 180° , as required by symmetry. The torsion angle $\text{O1}-\text{S1}-\text{C2}-\text{C3}$ is $81.0(2)^\circ$. The $\text{S1}-\text{O1}$ bond length [$1.495(2)\text{ \AA}$] is almost equal to that of the corresponding S—O bond [$1.487(2)\text{ \AA}$] of the analogous compound (*R,S*)-1,2-bis(phenylsulfinyl)ethane (Cattalini *et al.*, 1979), but the $\text{S1}-\text{C1sp}^3$ bond length [$1.799(3)\text{ \AA}$] is shorter than that of the S— Csp^3 bond [$1.814(4)\text{ \AA}$] found in (*R,S*)-1,2-bis(phenylsulfinyl)ethane.

Experimental

1,2-Bis(benzylthioether)ethane was prepared according to a procedure similar to that reported by (Hartley *et al.* (1979)). The oxidation of the bithioether to the corresponding disulfoxide was carried out according to the method of Zhang *et al.* (1995). Concentrated nitric acid (2.5 ml) was added dropwise to 5 g (0.016 mol) of 1,2-bis-(benzylthioether)ethane and stirred vigorously at room temperature for 24 h. The organic phase was separated from the resulting mixed solution and was washed with 10% sodium carbonate solution and water to separate out the solid product. Then the crude product was recrystallized from acetone. Yield: 45%, m.p: 493–495 K; IR (KBr pellets, cm^{-1}): 2963 (*m*), 1494 (*m*), 1455 (*m*), 1073 (*w*), 1019 (*s*), 1002 (*m*), 770 (*m*), 699 (*s*), 480 (*m*); $^1\text{H NMR}$ (CDCl_3): δ 2.83–3.04 (4H, *m*), 4.03 (4H, *t*), 7.26–7.40 (10H, *m*); analysis calculated for $\text{C}_{16}\text{H}_{18}\text{O}_2\text{S}_2$: C 62.66, H 5.92%; found: C 62.42, H 6.07%. Colorless single crystals were obtained by recrystallization from chloroform.

Crystal data

$\text{C}_{16}\text{H}_{18}\text{O}_2\text{S}_2$	$D_x = 1.343 \text{ Mg m}^{-3}$
$M_r = 306.42$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2123 reflections
$a = 16.933 (6) \text{ \AA}$	$\theta = 1.2\text{--}25.0^\circ$
$b = 5.312 (2) \text{ \AA}$	$\mu = 0.35 \text{ mm}^{-1}$
$c = 8.423 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 91.209 (7)^\circ$	Block, colorless
$V = 757.5 (5) \text{ \AA}^3$	$0.20 \times 0.20 \times 0.20 \text{ mm}$
$Z = 2$	

Data collection

Bruker CCD area-detector diffractometer	1333 independent reflections
φ and ω scans	994 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$R_{\text{int}} = 0.030$
$T_{\text{min}} = 0.933$, $T_{\text{max}} = 0.933$	$\theta_{\text{max}} = 25.0^\circ$
2943 measured reflections	$h = -20 \rightarrow 17$
	$k = -6 \rightarrow 5$
	$l = -10 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.130$	$(\Delta/\sigma)_{\text{max}} = 0.007$
$S = 1.11$	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
1333 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
91 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.25 (3)

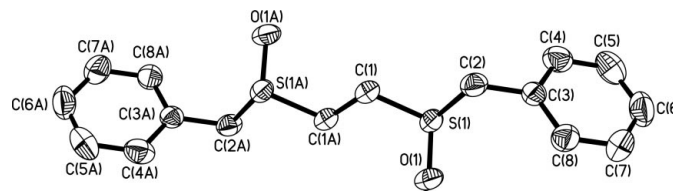


Figure 1

View of the title compound, showing ellipsoids at the 50% probability level.

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—O1	1.495 (2)	S1—C2	1.813 (3)
S1—C1	1.799 (3)		
O1—S1—C1	105.82 (14)	C1—S1—C2	97.63 (14)
O1—S1—C2	107.59 (15)		

H atoms were placed geometrically ($\text{C—H} = 0.93$ and 0.97 \AA) and included in the structure-factor calculations.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SHELXTL (Bruker, 1998); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

The authors gratefully acknowledge the financial support of the National Natural Science Foundation of China (No. 29971019) and the Tianjin Natural Science Foundation.

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